A STUDY OF THE EFFECTS OF FIN AND OVERLAP HEAT SEALS ON THE PERMEABILITY OF SELECTED PLASTIC FILMS

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ABSTRACT

A STUDY OF THE EFFECTS OF FIN
AND OVERLAP HEAT SEALS ON THE PERMEABILITY
OF SELECTED PLASTIC FILMS

by Richard F. Witte

Because permeability values of packaging materials may be affected by physical changes taking place during package fabrication, the author chose to study the effects of fin and overlap heat seals on the permeability of the material utilized for a packaging situation. The researcher studied the permeability relationships by utilizing a Davis cell and gas chromatographic analysis. Permeability determination was first carried out on the material. Fin and overlap heat seals were then applied to the same material, and permeability values were once again determined. Fin, overlap, and sample permeability values were compared for the same material. In this manner, the author not only determined the effects of heat sealing on permeability, but also the relative differences between fin and overlap style heat seals.

The major findings of the research pertain to the following materials: 2 mil nylon, 2.5 mil polyethylene/nylon laminant, 2 mil polyethylene/nylon/polyethylene laminant, 2 mil polyethylene/phenoxy/polyethylene laminant, 2 mil polyethylene. All results have shown that no significant changes occurred in the permeability rates due to fin and overlap heat seals.

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by

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Without the personal and professional support of Dr. H.E. Lockhart and David Pegaz, this work never would have achieved origin or substance. To these friends and to all who have contributed, my respect and my gratitude.

To

Sharon and Kristen

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Introduction

Although considerable research has been conducted regarding permeability of plastic films, very little work has been carried out regarding the effects of physical changes that may take place during package fabrication procedures. A typical physical change could be represented by the results of a heat sealing operation. Common laboratory procedures evaluate heat seal strengths, but such values may or may not have a bearing on the environmental performance of the seals later on in the total package life cycle.

The total package life cycle could be depicted by the following stages:

1. Material for package
2. Fabrication of material into a package
3. Package filling operations
4. Distribution of filled packages
5. Final purchase of package and product

When considering the total life cycle of a package, variables may occur within each stage resulting in changes in the overall performance of the package. In essence, it could be rationalized as moving from a known permeability value in the first stage to vague permeability awareness in the last stage. In addition, temperature, relative humidity, and time may all affect the package and contained product. However, since the proper film or packaging material can be matched against the most extreme conditions of environment in the distribution

channel, it becomes a problem of looking at the original package forming operations and resulting influences on the materials. It is known that package fabrication operations influence the material. The changes may affect the desirability of the product within the package. Because stages 3, 4, and ultimate satisfaction in stage 5 depend on stage 2, it becomes imperative to evaluate the effects of the variables taking place in stage two. The author chose to study the effects of fin and overlap heat seals on the permeability of the material.

Initial experimentation in permeability studies of heat seals on polyethylene indicated that a possibility did exist for an increase in permeability due to application of heat seals. The initial investigation led to a more refined technique for evaluating the effect of heat seals on the permeability of plastic films. The refined technique included the determination of leakage rates for fin seal, overlap seal, and material testing procedures as carried out with the Davis Cell. The Davis Cell coupled with gas chromatographic analysis enabled the author to determine the effects of fin and overlap heat seals on the permeability of the material.

Background

Brown(1)* in his article, "Permeability and Shelf Life", said that known permeability rates of flexible materials are unreliable guides for predicting the life of products contained within these materials. He concluded that package storage tests were the only reliable way of achieving guides.

Hu and Nelson(2) in 1953 experimented with water vapor and oxygen transmission through plastic films. At that time, they recognized that the integrity of the completed plastic package as in the case of a pouch could be severely damaged by the variability within the sealing processes. They expressed belief that a need for testing the effectiveness of the sealed area was of primary importance.

As early as 1944 and possibly earlier, Rabak and DeHority(3) experimented with the effects of heat sealing on the water vapor permeabilities of coated cellophanes. They believed that even though much work had been conducted on the water vapor resistance of packaging materials that were heat sealable, virtually no studies had been reported on the effects of sealing methods or the efficiency of the heat modified areas of the materials. They thought that the nature of the sealing operation had a direct bearing on the overall efficiency of the packaging material. Their initial hypothesis was as follows: If excessive temperature or pressures were utilized, it was quite possible to change the heat sealed material chemically as well as physically.

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*Numbers in parentheses identify Bibliography entries.

A reciprocal heat sealing device was used to control temperature, pressure, and time of contact. Sealing temperature, pressures, and times of contact were varied. The effects of the variables were determined by exposing the sealed samples to a standard dish water-vapor permeability test similar to the General Foods or Tappi Test Methods. The dish containing the sample was placed in a testing tunnel with an air velocity of 500 ft./minute, relative humidity at 87%, and an average temperature of 89°F. The vapor pressure differential between the atmosphere in the tunnel and the inside of the test dish was calculated to be 28 mm of mercury. Calculation of the water vapor permeability constants was expressed as grams of water vapor passing through one square meter of surface per 24 hr. / mm of mercury difference in water-vapor pressure between the outside and inside of the dish.

Two heat seal treatments were used: 1) an imprint treatment to permit measurement of the effects of temperature and pressure on the lacquers of single thicknesses of cellophane and, 2) overlap seals were included to ascertain the extent of edge leakages. The following conditions were used for heat sealing purposes:

Sealing time -- 1 second

Sealing temperature -- 285° - 385° - 450°

Pressures -- 10 and 50 p.s.i.

The results of the tests indicated definite trends:

1. Sealing temperatures of 285°F were not high enough to create firm bonds.

2. Sealing temperature of 385°F. created firm bonds with considerable disturbance of the lacquers resulting in permeability increases of 3 to 8 times over that of the material.
3. Temperatures of 450°F. were very destructive.
4. Ten p.s.i. caused considerably less impairment than 50 p.s.i..
5. The efficiency of overlap seals appeared to be less affected by variations in sealing pressures than the imprint type.

Rabak and Stark(4) developed a starch-iodide method that demonstrated the porosity of the heat seal area of waxed paper. The method consisted of passing a heat sealed waxed wrap through a 1% aqueous iodide solution. Subsequent to this, the wrap was washed in water and dipped in a 1% starch solution. Deposition of blue-black starch iodide within the heat sealed area served as an indicator of the porosity of the heat sealed area.

Rabak and Stark(5) also experimented with sealing temperature effects on waxed paper regarding water vapor permeability. By varying heat sealing temperatures on various types of coatings coupled with the Tappi Dish Determinations Method, they concluded that heat sealing definitely impaired the water vapor resistance of waxed papers. The extent of impairment increased with the elevation of sealing temperature.

Other seal tests performed by researchers included those of Edwards and Strohm(6). Their main concern involved the

efficiency of seals on foil packages. They could see little value in utilizing impervious wrapping materials while leaving the joints unsealed in such a manner as to permit ready movement of air in and out of the package. Packets made from foil were tested for water vapor permeability by enclosing anhydrous calcium chloride in a heat sealed package. The packets were stored in a cabinet at 100°F. and 90% R.H. Nine months were then allowed to pass and the increase in weight was recorded. The efficiency of various closures was investigated by tests on cartons and bags fabricated in several ways and sealed by different methods. The results indicated that aluminum foil with applied heat seal proved to be the best method of closure.

Past records indicate that substantial research has been conducted on seal integrity regarding water vapor permeability. The latest trend has been to test the entire package in a pouch or similar form. This is the next most logical step to further package research.

Brickman(7) measured the gas transmission rates of materials in pouch form. He also expressed doubts regarding permeability values of materials in sheet form due to the non-representative exposure conditions. The method used by Brickman simulated actual package conditions better than other techniques. His method consisted of a pouch formed over a styrene insert. The insert was utilized to assure a nearly uniform volume for the pouch. The air was exhausted from the pouch, and nitrogen was introduced at a specified gauge pressure. A rubber patch cemented on the side of the pouch

facilitated syringe samples without distorting the pouch in any manner. Gas samples were drawn off from the pouches and introduced into a Beckman Gas Analyzer. Pouch conditions included wet and dry interiors exposed to different room and refrigerated temperatures and relative humidities. Duration of tests for laminated materials of low oxygen permeability was from 20 to 30 days. This was necessary to allow equilibrium or a fairly constant permeation rate. The bulk of Brickman's work was concerned with cellophane/polyethylene, mylar/polyethylene and other forms of similar laminants. The following is a summary of Brickman's results:

1. Cellophane/polyethylene pouches were affected more by changes from dry to wet packs than other laminants in terms of permeation.
2. Pouches formed of laminated materials were not affected nearly as much by changes in exposure conditions as polyethylene and mylar materials.
3. Polyethylene wet packs had higher oxygen permeation rates than did dry packs exposed to the same conditions.

Experimental Equipment and Procedures

The permeability testing system is shown by the schematic in Figure I. The system was developed by Lockhart(8). Oxygen and nitrogen were the two gases utilized for all tests. Tests were conducted under "dry conditions"; consequently, no humidity devices were required within the system itself. The humidity sensing device was used only for the assurance of dry gas conditions. Dry conditions were represented by 1.5% relative humidity or less. The relative humidity sensing device was an Electric Hygrometer Indicator (Catalog no. 4-4902) by American Instrument Company with Aminco-Dunmore sensing elements.

A manometer within the system permitted detection of any differential pressure in the Davis Cell. The sweep gas, nitrogen, and the testgas, oxygen, both pass through drying tubes that contain Drierite Dessicant. Flow measurement was achieved with rotameters by Brooks and Fisher-Porter and metering valves by Hoke(8). As mentioned previously, the manometer permitted visual examination of the pressure differential between the sweep and test gas lines. The differential was zero during all tests; thus achieving isostatic conditions within the Davis Cell.

The Davis Cell is stainless steel, constructed in two sections. It provides a sample area of 156 square centimeters (Figure 2). The cell is locked in a clamp which applies pressure at three points on the top of the cell. The seal between the upper portion of the cell and the material to be

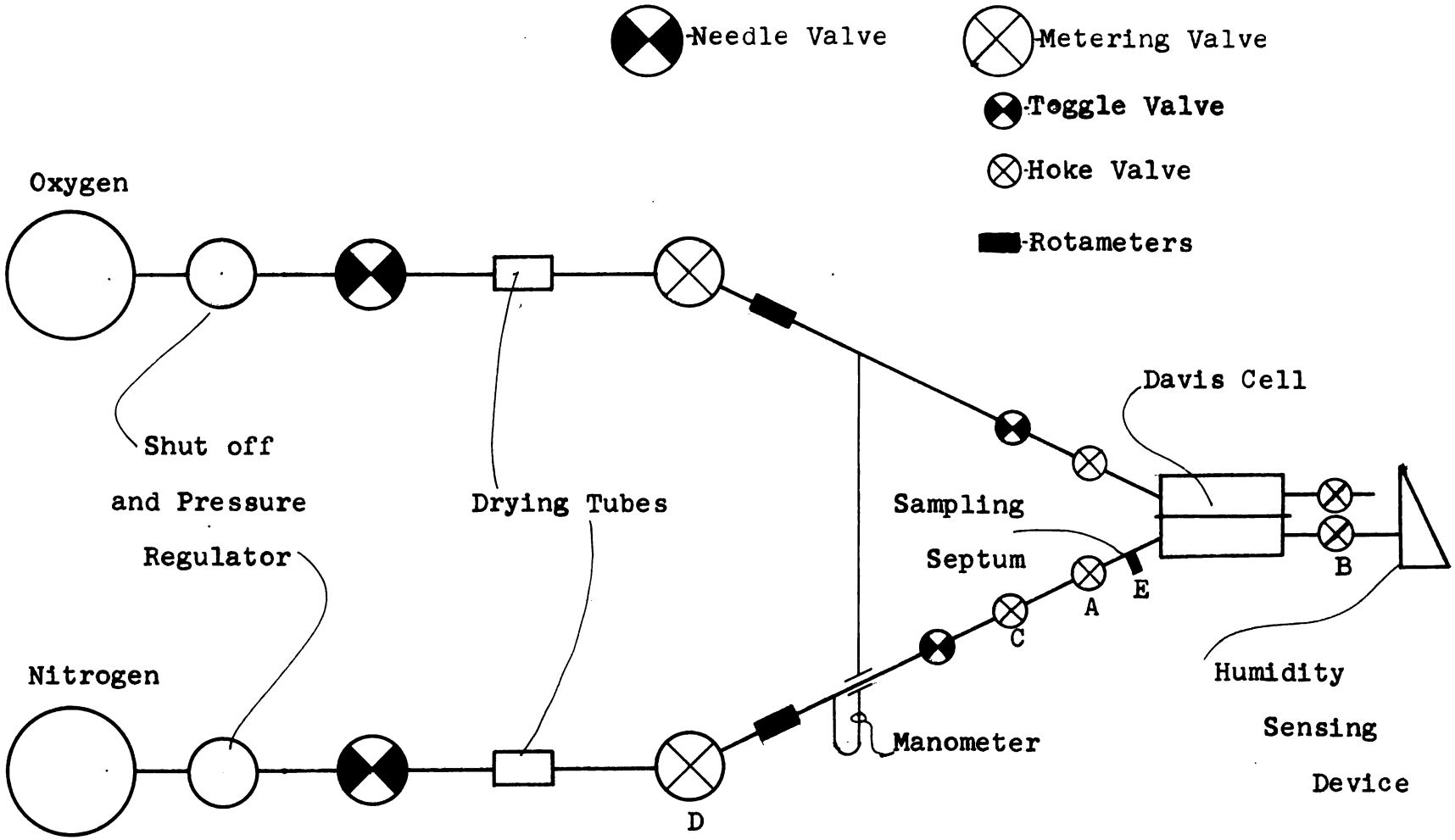

Figure 1

Permeability Testing System

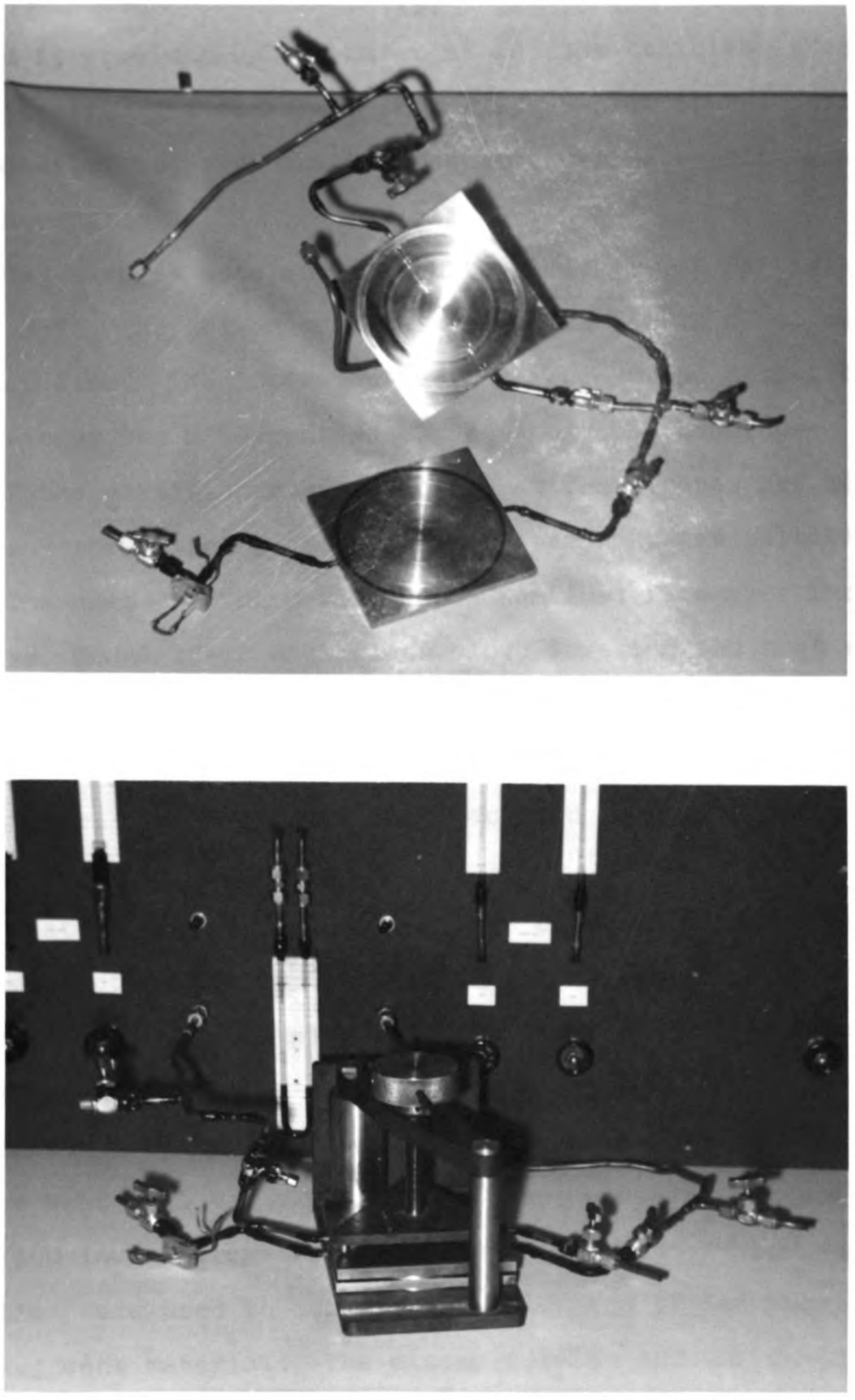

Figure 2

Davis Cell in Open(top) and Closed Positions

tested is provided by the material and the stainless steel cell itself. The seal between the lower half of the cell and the material is provided by a neoprene O-ring bearing against the material.

Gas samples were analyzed with a Fisher Gas Partitioner, Model 25V. The column system is designed for an 80 ml/minute carrier flow. The instrument used to record the output of the partitioner was a Sargent Model SR Laboratory Recorder. Output of the partitioner was recorded at four inches per minute. Figure 3 shows the recorder, cell system, and gas partitioner.

The heat sealing device was a Sentinel Pacemaker Thermal Impulse, Model 12-TP with glass cloth covering the heat sealing bands on both jaws (Figure 4). Jaw pressures were controlled by an air pressure valve connected to a pressurized line within the sealer. Total impulse range covered 1.20 seconds in graduations of 0.10 seconds.

Heat sealed cross sections were observed with a Scherr Microprojector. A 100 magnification lens was used for all observations. The samples were held in a device so as to facilitate exposure of the edge of the heat seals to the light and lens. Seals were viewed on a screen upon which the image of the heat seal was reflected. A steel machinist's scale in 1/100 inch increments and a small hand lens of ten magnification were used to measure the thickness of the heat seals and adjacent material. The microprojector and the device for holding the samples are shown in Figure 5.

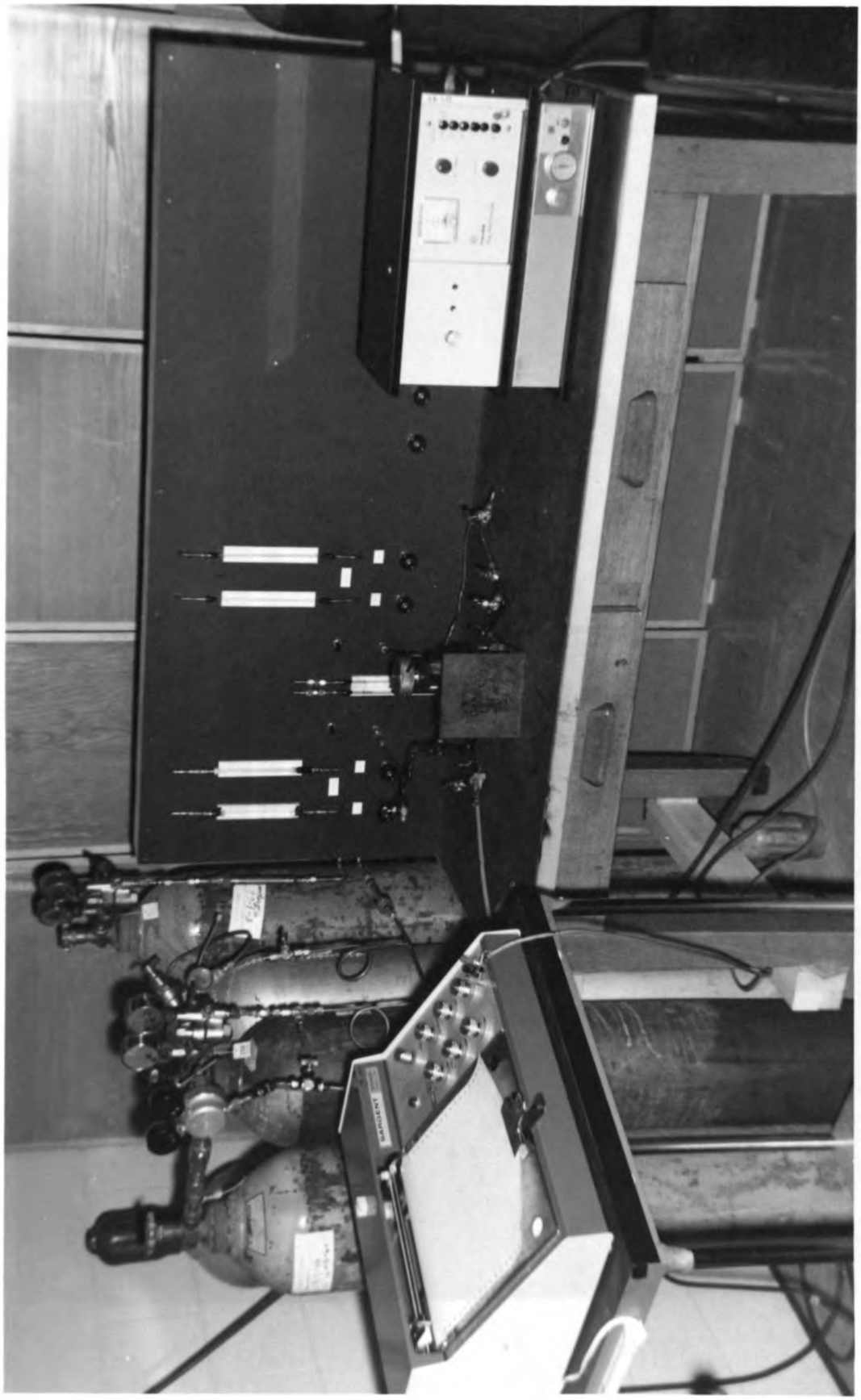

Figure 3

Recorder, Cell System and Gas Partitioner

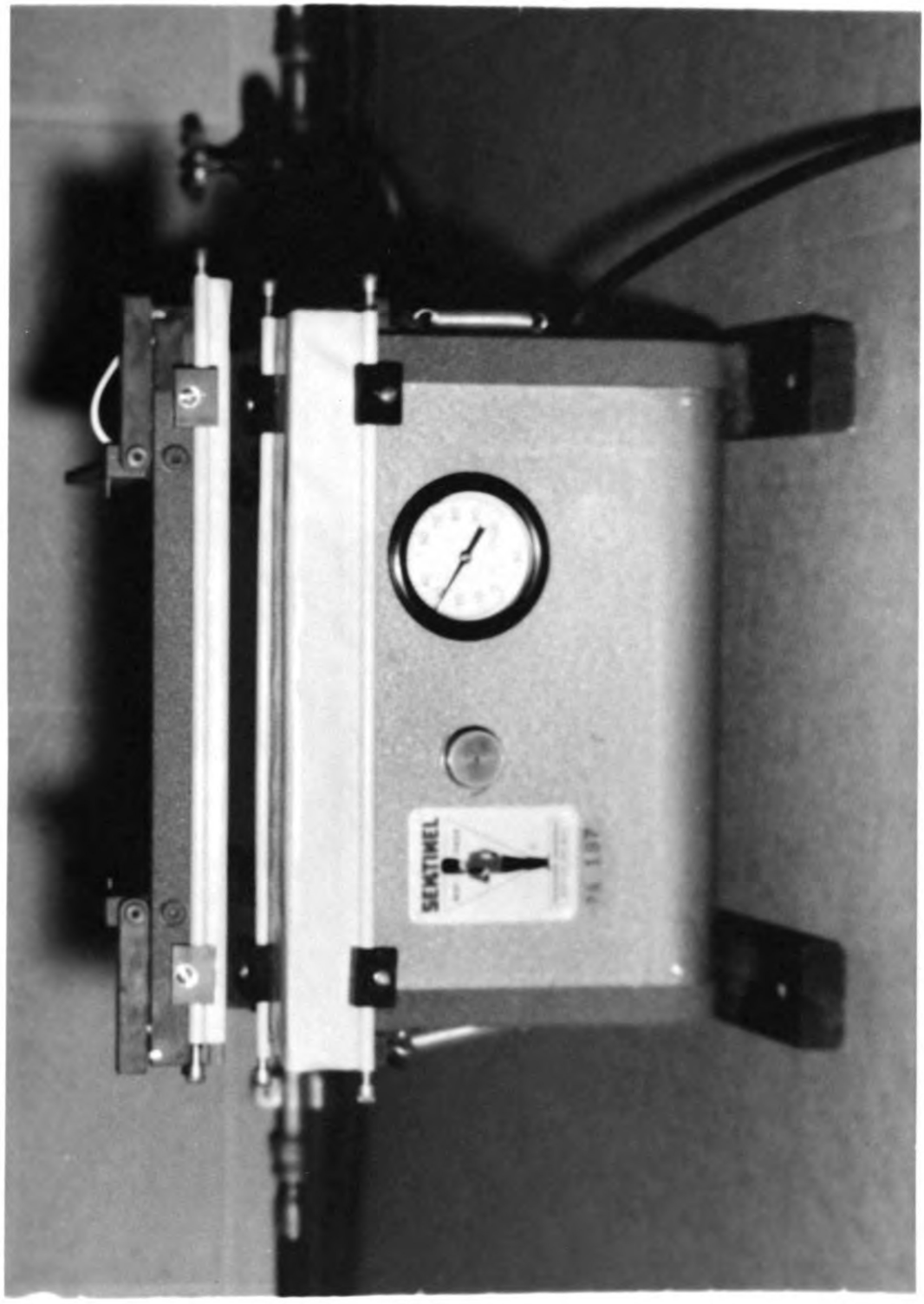

Figure 4

Sentinel Impulse Heat Sealer

Figure 5

Scherr Microprojector and Device to hold Heat Seals

Earlier tests had indicated an excessive leakage rate for the Davis Cell. It, therefore, became necessary to examine the valves on the lower half of the cell to determine the source of the leak. The original valves were identified as the source of the leak, and new valves were installed. Valve leakage determination is explained in Appendix I. The following description of testing procedures helps to explain why various components of the cell system were leak rated. The film sample to be tested was placed in the cell with a stainless steel-sample seal on the top half and an O-ring-sample seal on the bottom half of the cell. After the film sample was placed in the cell, a 100 ml/min. oxygen and nitrogen flow was imposed on each side of the cell. Both sides were allowed to flow for one-half hour. After this period of time, the bottom half of the cell was isolated by means of the two Hoke valves. The cell system would then be as shown in Figure 6.

The permeation rate of oxygen into the lower half of the cell was determined by taking samples from the lower half with a syringe and septum device. All samples were drawn from the lower half of the cell; consequently, the leakage rate between the film sample and the O-ring had to be determined for final calculation of permeability rates.

After the leakage rate determination of the new valves, the valves were installed on the lower half of the Davis Cell. Subsequent to this, a leakage rate determination was carried out for the cell system. The lower half cell-valve system was leak tested as shown in Figure 6 with aluminum foil serving as a blank. This test gave a true indication of the leakage rate

⊤- Hoke Valve (closed)

•- Sampling Septum

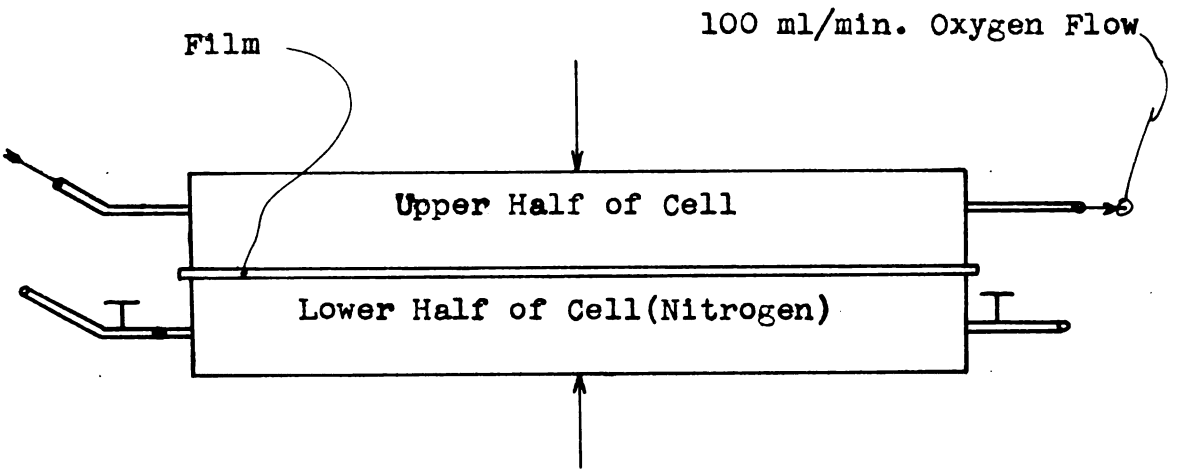

Figure 6

Davis Cell in Testing Simulation

between the O-ring on the bottom half and the aluminum blank. Due to the physical differences of fin and overlap seals, it was necessary to leak test both heat seal styles. It was also necessary to study the gap distance between cell faces if constant leakage rates were to be established. Earlier experimentation of leak testing fin and overlap seals without a known gap distance had introduced a problem of obstruction of oxygen and nitrogen flows. The force applied in closing the cell could be such that the interior clearance between top and bottom of the cell was reduced to zero or nearly so. At that minimum point, gas flows across the surfaces of the film with fin or overlap seals were obstructed. The problem involved minimizing the leakage rates for fin and overlap seals while arriving at a maximum gap distance for proper gas flows. Figure 7 shows the gap distance (x) arrived at by a trial and error method. The trial and error method established leakage rates at decreasing gap distances using aluminum blanks(Table I). After test number 8 (Table I) had been performed, an overlap seal-aluminum blank combination was tested in the configuration shown in Figure 8. At this time, it was discovered that a 0.010 inch gap distance was the minimum distance that could be used for proper gas flows. Once the correct gap distance was established, only fin seal-aluminum blank combinations remained to be leak rated. All heat seals were perpendicular to the flow of the test gas. This was true for leakage rate determination and actual material testing procedures. All material testing was done with a 0.010 inch gap distance.

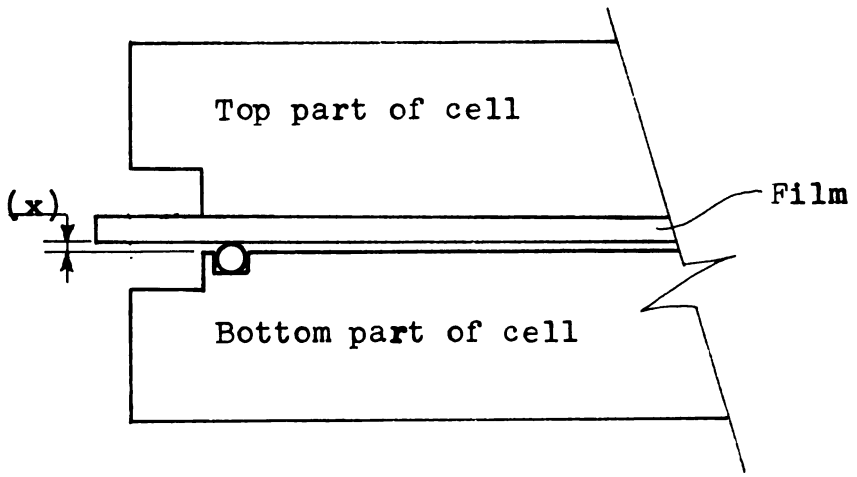

Figure 7

Cell Gap Distance

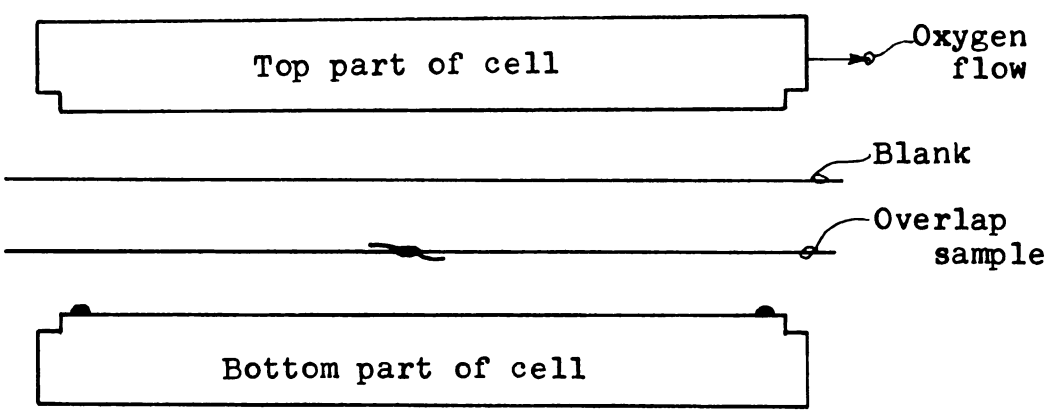

Figure 8

Aluminum Blank and Overlap Sample Position

Table I. Davis Cell Leakage Rate Tests

Test No.	Type of Test	Gap Distance	Leakage Rate (ml/24 hr.-m^2)
1	2.5 mil aluminum blank	Unknown	2
2	2.5 mil aluminum blank	Unknown	2
3	Installed new aluminum blank	Unknown	5
4	Aluminum blank with 1 mil nylon overlap heat seal under the blank	Unknown	9
5	Same as no. 4 - new samples installed	Unknown	9
6	Aluminum blank with 1 mil nylon overlap heat seal under the blank	0.016	81
7	" " "	0.013	80
8	Aluminum blank	0.010	5
9	Aluminum blank with 1 mil nylon overlap heat seal under the blank	0.010	4
10	Aluminum blank with 2 mil nylon overlap heat seal under the blank	0.010	4
11	Aluminum blank	0.010	13
12	Aluminum blank	0.010	10
13	Aluminum blank with 2 mil poly/phenoxy/poly fin heat seal under the blank	0.010	12
14	Aluminum blank with 2 mil nylon fin heat seal under the blank	0.010	5

The highest leakage rate was 13 ml/24 hr.-m^2; the lowest rate was 2 ml/24 hr.-m^2 (Table I). Overlap and fin seals probably did not contribute to a much higher rate. The leakage rate proved to be very low regardless of the conditions. The author chose to use an average of all leakage values in tests 8-14. Tests 1-5 were not included due to the unknown gap distances. Tests 6-7 were excluded because of the obviously incorrect gap distances. The calculated average leakage rate was 7 ml/24 hr.-m^2.

Leakage rate studies indicated that the number of samples extracted from the cell had a direct bearing on the apparent permeability. It was also discovered that errors would be introduced when intervals between samples were one hour or less. It was decided not to take more than six to seven samples at intervals of 1 1/2 to 2 hours. The sampling intervals were dependent on the material being tested; only 2 mil polyethylene with its high permeability rate required sample intervals of less than one hour. Tests conducted overnight were based on three to four samples. Syringe samples were 0.20 ml. Total volume extracted from the cell was 1.20 to 1.40 ml. per test. This represented approximately 6% of the cell volume from which the samples were drawn.

The materials used for permeability testing were:

1. 2 mil nylon 6-Capran, type 77c
2. 2.5 mil polyethylene/nylon lamination
    a. 1.5 mil Capran
    b. 1.0 mil polyethylene

3. 2 mil polyethylene/nylon/polyethylene lamination
    a. 1.0 mil nylon
    b. 2 - 0.5 mil polyethylene
4. 2 mil polyethylene/phenoxy/polyethylene lamination
5. 2 mil polyethylene - Dow Poly Film, type 114 t-1

The permeability of a chosen material was first established by conducting two tests on two different samples. In all cases, agreement between the two samples proved to be very satisfactory. After establishing the permeability of the material, heat sealed samples were exposed to the same conditions to determine the effect of the heat seal. Refer to Appendix II for the permeability testing procedure and Appendix III for permeability value calculations.

A suitable heat sealing combination was difficult to establish for 2 mil nylon material. Pinholes in the heat seal seemed to be the main problem. It was decided to avoid wasting "cell time" by fabricating sealed samples of the material for heat seal integrity testing prior to installation of another like sample for permeability testing. The heat seal integrity test was very simple, took little time, and required no additional equipment. It was performed as shown in Appendix IV. Overlap heat sealed samples of 2 mil nylon material were pressure tested over the following impulse-pressure settings: Impulse times were varied from 0.10 seconds to 1.20 seconds in 0.10 second intervals. Pressure was maintained at 15 p.s.i.. All results indicated "leakers". Higher impulse-pressure combinations were attempted. A successful combination of 0.80

sec.-60 p.s.i. produced suitable seals. Heat seal combinations for the remaining four materials were much simpler to establish. Adoption of the heat seal integrity test enabled the author to initiate permeability testing as close as possible to the heat seal leakage boundaries of the last four materials. The heat seal leakage boundary of a particular material-impulse-pressure combination was approached as follows: Pressure was held constant; impulse times were varied in even increments from low to high settings. Each successive heat seal was subjected to the heat seal integrity test. If it passed the test, another heat seal was fabricated with a higher impulse setting. Eventually a point (boundary) was reached where the material-impulse-pressure combination produced heat seals that failed the seal integrity test. Permeability testing was conducted on the heat sealing combination that immediately preceeded the combination that failed. Possible distortion factors could change the characteristics of the material while undergoing the seal integrity test. In no cases were one and the same sample subjected to the seal integrity and permeability test.

Standardizing all techniques associated with the experimental equipment and procedures made it possible to produce precise and valid results. The reported values for leakage and material testing represent results that were achieved by careful consideration of the variables that were treated as constants after study.

Experimental Results

Regardless of the material, all heat seal cooling times were maintained at 6.0 seconds. No mention of cooling times are made in the remaining text. All impulse and jaw pressure variables are shown as seconds - p.s.i..

Two mil nylon material was used for initial permeability testing. After establishing the permeability rate of the material, overlap and fin style heat seals were applied to the material with a 0.80 sec. - 60 p.s.i. combination. The permeability values of the unsealed material were compared to the values of the heat sealed material (Table II). As the results indicate, no apparent significant differences exist.

The first laminated material selected for study was 2.5 mil polyethylene/nylon. Two mil nylon had not shown variability in permeability with applied heat seals; therefore, it was decided to study the permeability of the approximate identical material in laminant form. The purpose was to reduce the pressure or impulse and contain the entire depth of the heat seal within the polyethylene coating.

Two mil polyethylene/nylon/polyethylene and 2 mil polyethylene/phenoxy/polyethylene were the last laminated materials tested. All values are shown in Table II.

Preliminary experimentation with overlap heat seals on polyethylene had indicated a possible increase in permeability. The data in Table II for 2 mil polyethylene seemed to substantiate the preliminary experimentation. To assure proper analysis, data of a different nature was gathered. Heat seal

Table II Permeability Values of all Samples

Material	Aggregate Perm. Rate (ml/24 hr.-m^2)	Leakage Rate (ml/24 hr.-m^2)	Actual Perm. Rate (ml/24 hr.-m^2)	Av. Perm. Rate (polyethylene) (ml/24 hr.-m^2)	Heat Seal Style or Material	Impulse-Pressure (Sec.-p.s.i.)	No. of Heat Seals Per Sample	Total Heat Sealed Area(m^2)	Heat Seal Area Total Sample Area (%)	No. of Tests
2 mil nylon	23	7	16		Material					Average of 2
"	21	7	14		Overlap	0.80 - 60	1	.000448	2.87	Average of 2
"	20	7	13		Fin	0.80 - 60	1			1
2.5 mil poly/nylon	40	7	33		Material					Average of 2
"	38	7	31		Overlap	0.50 - 20	1	.000448	2.87	1
"	38	7	31		Overlap	0.75 - 20	1	.000448	2.87	1
"	38	7	31		Fin	0.75 - 20	1			Average of 2
2 mil poly/nylon/poly	63	7	56		Material					Average of 2
"	62	7	55		Overlap	0.45 - 15	1	.000448	2.87	1
"	61	7	54		Overlap	0.35 - 15	1	.000448	2.87	1
"	63	7	56		Fin	0.35 - 15	1			Average of 2
2 mil poly/phenoxy/poly	203	7	196		Material					Average of 2
"	204	7	197		Overlap	1.00 - 20	1	.000448	2.87	Average of 2
"	205	7	198		Overlap	0.25 - 20	1	.000448	2.87	1
"	198	7	191		Fin	1.00 - 20	1			Average of 2
2 mil polyethylene	2293	7	2286	> 2.2%* - 2261	Material					1
"	2243	7	2236		Material					1
"	2308	7	2301	> 1.8% - 2322	Overlap	1.00 - 30	1	.000448	2.87	1
"	2350	7	2343		Overlap	1.00 - 30	1	.000448	2.87	1
"	2243	7	2236		Overlap	1.00 - 30	2	.000877	5.68	1
"	2386	7	2379	> 0.60% - 2385	Overlap	1.00 - 30	4	.001710	10.96	1
"	2399	7	2392		Overlap	1.00 - 30	4	.001710	10.96	1
"	2258	7	2251		Overlap	1.00 - 30	7	.002913	18.6	1
"	2343	7	2336		Fin	1.00 - 30	1			1

*Indicates the spread for two tests based on the larger value

thickness measurements (Table III) provided the additional information. Two mil nylon had not shown significant permeability value differences. Because of this, the researcher wanted to compare heat seal thickness measurements for nylon and polyethylene material. Final analysis would then depend on the comparison of the data in Table II and III.

Table III Microprojection Measurements of Polyethylene and Nylon Heat Seals (100 Magnification)

Material	(1) Dbl. Thickness of Material (inches)	(2) Seal Thickness at Center (inches)	(3) Imp.-Pres. (Sec.-p.s.i.)	(4) Column 2 / Column 1	(5) The Average (%)
2 mil nylon	0.440	0.400	0.80-60	90.9	
2 mil nylon	0.450	0.425	0.80-60	94.4	
2 mil nylon	0.450	0.425	0.80-60	94.4	92.9
2 mil nylon	0.450	0.420	0.80-60	93.2	
2 mil nylon	0.480	0.440	0.80-60	91.7	
2 mil polyethylene	0.460	0.330	1.00-30	71.7	
2 mil polyethylene	0.450	0.330	1.00-30	73.3	
2 mil polyethylene	0.450	0.335	1.00-30	74.4	73.6
2 mil polyethylene	0.460	0.340	1.00-30	73.9	
2 mil polyethylene	0.455	0.340	1.00-30	74.7	

Analysis and Major Findings

Extensive preliminary experimentation was required to become familiar with the testing system. Air samples at 72°F. -50% relative humidity were introduced into the chromatographic system to determine total experimental error. The following variables were considered during experimental error determination: Chromatographic response, recorder response, gas sampling procedures and operator variation during the sampling procedure. Total experimental error amounted to 6 percent.

Research of this nature requires standards for comparison purposes. The standards are represented by the permeability values of the unsealed materials. If a heat sealed sample permeability value differed from the standard (unsealed) by ±3% or less, it was reasonable to assume that heat sealing has no apparent effects on permeability rates. Applying this to 2 mil nylon, Table II shows a range from 16 ml/24 hr.-m^2 for the unsealed material to 13 ml/24 hr.-m^2 for the fin style heat sealed material. The 3 ml/24 hr.-m^2 difference represents a-18.7% (3/16) variation or approximately six times the experimental error. Further analysis was required before final conclusions were reached. The first two materials have very low permeability rates. Experimental error and leakage rates have a far greater effect on the outcome than for those materials with higher permeability rates. To demonstrate, the leakage rate is approximately 33% of the aggregate permeability rate for 2 mil nylon. The same leakage rate is only 0.31% of the aggregate permeability rate for 2 mil polyethylene. In tests

8-14(Table I) the average of the three aluminum blank tests is 9 ml/24 hr.-m^2. The average for all overlap and fin tests in the same grouping is 6 ml/24 hr.-m^2. Referring to 2 mil nylon(Table II) the aggregate permeability rate of the material -the new leakage rate (23 ml/24 hr.-m^2-9 ml/24 hr.-m^2) = the aggregate permeability rate of the fin style - the new leakage rate (20 ml/24 hr.-m^2 - 6 ml/24 hr.-m^2). This demonstrates the insignificance of the small differences that are shown in Table II for two mil nylon.

To summarize, the -18.7% difference for 2 mil nylon and the -6.1% difference for polyethylene/nylon laminated material does misrepresent the facts. The insignificance of the small differences for the first two materials and total test equipment limitations dictate the only possible conclusion: Permeability rates have not been affected by fin and overlap heat seals.

Analysis of the next two materials is much simpler. The leakage rate is approximately 11% of the aggregate permeability rate for polyethylene/nylon/polyethylene and 3.5% for polyethylene/phenoxy/polyethylene. The leakage rate is less significant for final analysis of the two materials. Table II shows a range from 56 ml/24 hr.-m^2 (unsealed) to 54 ml/24 hr.-m^2 (sealed) for 2 mil polyethylene/nylon/polyethylene. Two ml/24 hr.-m^2 represents a -3.5% difference. Likewise for 2 mil polyethylene/phenoxy/polyethylene, the 198 ml/24 hr.-m^2 rate and the 191 ml/24 hr.-m^2 rate represent a +1% and a -2.5% difference. The only possible conclusion is that permeability

rates have not been affected by heat sealing.

Analysis of 2 mil polyethylene can be done in one of several ways:

1. Compute an average of all heat sealed samples and compare with the standard.
2. Compute an average of all overlap sealed samples and compare with the standard.
3. Compute group averages and compare with the standard.

Permeability values used in defining the ranges for the first four materials consisted of the average of two tests. Values for 2 mil polyethylene under the same testing conditions seemed to differ by a larger amount. Because of this, each individual test value is shown and not the average of two tests. Maintenance of continuity was of extreme importance. Methods 1 and 2 were not used because each involved values that had not been duplicately tested regarding number of heat seals per sample. Group averages were computed (column 5); the range for the unsealed and sealed samples was 2261 - 2385 ml/24 hr.-m^2. The difference (+124 ml/24 hr.-m^2) was +5.5%. A possible increase could have occurred. Further analysis nullifies the possibility. Permeability rates did not increase as the total area exposed to heat seals increased (Table IV). The rates fluctuated, and in two cases (2 overlap and 7 overlap) the rates approached the average unsealed rate. In addition, no significant changes in permeability rates were noted as the ratio of heat sealed to total exposed area increased approximately 2, 5 and 9 times.

Table IV Permeability versus Number of Heat Seals on Polyethylene

Actual Perm. Rate ($ml/24\ hr.-m^2$)	Number of Overlap Heat Seals per sample
2261*	none(material)
2322*	1
2236	2
2385*	4
2251	7

*Average of two tests

Microprojection analysis (Table III) substantiates the permeability results. Table III shows that the average (%) seal thickness/double thickness of material for polyethylene was 73.6. The seal thickness was 1 mil greater than the thickness of the material undergoing the test. Because the heat sealed area was 1 mil thicker than the unsealed area, a possible lower overall permeability rate could have occurred. The results in Table II do not substantiate this idea. Therefore, the overall affect of the thickness increase in the heat sealed portion was probably negated by the physical changes occurring at the time of the heat seal. Materials utilized for the tests were generally oriented. Upon application of heat, the heat sealed area consisted of an amorphous condition. Cooling effects crystallized the seal area into a non-oriented matrix of random pattern. The physical change from oriented to non-oriented pattern may have increased the permeability rate by approximately the same amount as the seal thickness decreased the rate. The end result being a negation of the two variables.

The prior analysis is highly speculative. It may be asked: Why did the author in the case of polyethylene not try to utilize a heat sealing combination that would have resulted in a seal thickness less than 2 mils? This is justified by the practical approach to packaging. Why utilize a heat sealing combination producing such an effect when in all probability it would never be used in a packaging situation. Based on the prior analysis of all tested materials, the following list indicates the major findings of the study:

1. No apparent permeability differences exist between sealed and unsealed materials.
2. No apparent permeability differences exist between heat seal styles (fin and overlap).
3. No apparent permeability differences exist between laminated and non-laminated heat seals.

Discussion and Conclusions

The entire project was approached on the basis of a general formula. The general formula was: Permeability rate of the unsealed material + the permeability rate of the heat sealed area on the same sample = the total permeability rate of the sample. By varying the area of exposure to heat seals, which in turn varied the unsealed area on the same sample, it would have been possible to arrive at a permeability rate for given conditions. The general formula would then be used as a method of permeability prediction for package fabrication. After considering all test results, no significant figures could be plugged into the general formula.

As discussed in Experimental Equipment and Procedures, 2 mil nylon material was very difficult to heat seal. Most highly impermeable materials do present heat sealing problems. This is why materials with good heat sealing characteristics are laminated to highly impermeable (barrier) materials. The resulting hybrids have good heat sealing qualities coupled with low permeability rates. After considerable experimentation with 2 mil nylon, a successful heat sealing combination was achieved. Any apparent deviation outside of the combination resulted in poor heat seals. Herein lies the most prominent reason for laminated forms of barrier materials: Mechanical and electrical deviations will occur within the mechanism controlling heat sealing operations. The deviations result in temperature and pressure differences that fall outside of the acceptable heat sealing range, creating poor heat seals.

Coating the barrier member with a material capable of absorbing a wide heat sealing range solves the deviation problem. The coating material prevents burning or searing of the barrier material and allows lower impulse and pressure settings. Lower impulse and pressure settings are advantageous; they result in less wear on heat sealing mechanisms.

The author intended to study heat sealing effects on non-laminated barrier materials. At the same time it was realized that no evidence existed regarding the permeability of laminated heat seals versus the permeability of non-laminated heat seals. Laminated barrier materials account for a large portion of the total plastic packages on grocery shelves today. A practical approach to the research was necessary to study laminated and non-laminated materials for comparison purposes.

While conducting preliminary research, evidence indicated that impulse variations did not change the permeability rates of heat seals. This isshown for a few samples in Table II. It was felt that impulse should have effected permeability more than pressure, consequently; pressure variations were not studied.

It is the opinion of the author that heat sealing effects do not produce any apparent significant variations in material permeability. Future related package research should be concentrated in the following areas:

1. Additional research should be conducted on laminated barrier materials to establish the relationship between permeability and physical strength of heat seals.

2. Research should be conducted only on heat seal areas of films with known crystalline orientation to determine the following:
   a. The relationship of the physical changes that take place during heat sealing and how the changes affect permeability.
   b. The permeability-thickness relationship of heat seals.
   c. How humidity influences heat seal permeability.

APPENDICES

Appendix I

Valve Leakage Determination

The two Hoke Valves on both sides of the bottom half of the Davis Cell were suspected of contributing a large part of the total leakage rate. The two valves were removed from the cell and installed on a 3/4 inch copper pipe. Total volume of pipe and valves approximated 25 ml. The pipe-valve system was attached to a nitrogen source, flushed, and sealed off in the same manner as the total cell system. Oxygen leakage through the valves was calculated by taking samples using the syringe-septum device. The testing device is shown in Figure 9. The valve testing procedure was:

1. Flush the system
2. Close valve A; close valve B
3. Turn off nitrogen and release pressure
4. Take gas sample from system
5. Introduce gas sample into gas chromatograph and analyze
6. Repeat 4 and 5 until a definite trend is noted

The syringe was flushed with nitrogen prior to sampling from the pipe-valve system. The end of the syringe needle was covered with a spare septum during transfer from the test fixture to gas chromatograph.

The results of the initial valve test indicated a high leakage rate on the order of 50 ml/24 hr.-m^2. New Hoke Valves were installed on the pipe and the same steps were repeated. The new valves proved to have an average leakage rate of 2 ml/24 hr.-m^2.

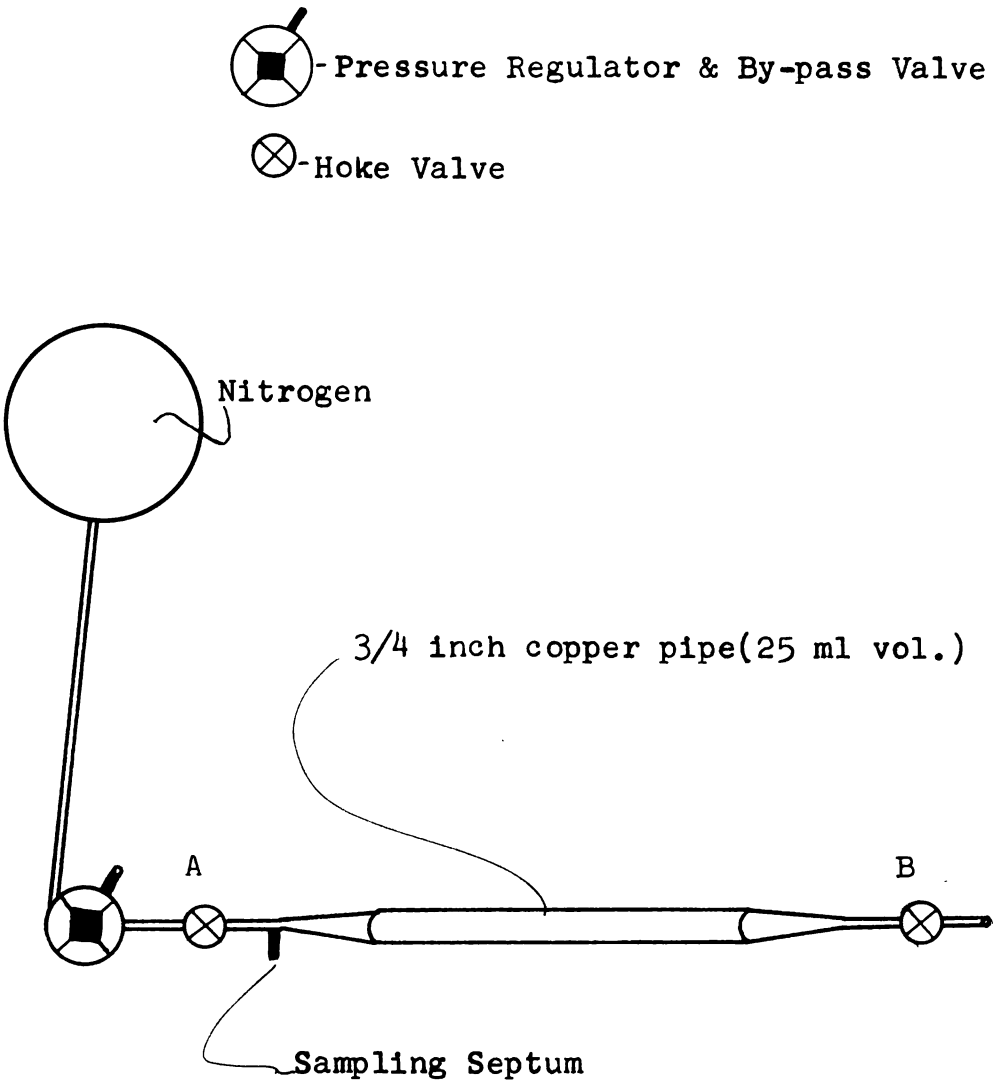

Figure 9

Valve Leakage Determination- Apparatus

Appendix II

Permeability Testing Procedure

Step 1 Attain helium carrier flow of 80 ml/min. in partitioner. Allow to flow for 15 to 30 min. Turn on recorder and adjust to zeroing position.

Step 2 While Step 1 was carried out, the sample to be tested was fabricated.

Step 3 After the period of 15 to 30 min., a 0.20 ml sample of air was injected into the partitioner for air factor determination.

Step 4 Place the sample to be tested in the cell.

Step 5 Clamp the cell shut holding the cell gap distance to 0.010 inches.

Step 6 Initiate 100 ml/min. test and sweep gas flows; allow 30 minutes to equilibrate.

Step 7 Isolate the bottom half of the cell by the following procedures: Refer to Figure I.

a. close valve A

b. immediately close valve B and open bypass valve C to prevent pressure build up from forcing glycerine throughout system

c. mark isolation time

d. turn off metering valve D and sweep gas source

Step 8 Allow test gas to flow at 100 ml/min. rate for duration of test; checking the flow periodically.

Step 9 Purge syringe in nitrogen bath and inject needle of syringe in septum E. (Figure I)

Step 10 Inject initial sample into the partitioner, mark the time, and turn on the recorder.

Step 11 Continue taking gas samples of 0.20 ml at intervals of 1, $2\frac{1}{2}$, 4, $5\frac{1}{2}$, and 7 hours. More samples were extracted if the previous points did not define a definite trend.

Appendix III

Permeability Value Determinations

Calculation of permeability rates and leakage rates were based on the following:

1. Area of material exposure to test gas - 0.015606 sq. meter
2. Volume of lower portion of cell - 20.59 ml

The oxygen rate of permeation was determined from the output of the Fisher Partitioner by means of the Sargent Model SR Recorder. Oxygen permeation was then expressed as a percentage per 24 hr. period. Calculation of oxygen percentage per sample was carried out in the following manner (Figure 10):

Y measured at a distance of X/2 for each trace.

(X) (Y) (Attenuation factor) = area for each trace.

In Figure 10, the appropriate attenuation factor for nitrogen is 50 and 1 for oxygen.

Percentage of oxygen was then expressed as a portion of the total sample designated by the sum of both graphs:

In the preceding example, let X = 10, Y = 1 for nitrogen

let X = 5, Y = 0.50 for oxygen

Area of nitrogen - (10) (1) (50) = 500

Area of oxygen - 5 (.50) (1) = 2.50

% oxygen increase $\frac{2.50}{500+2.50}(100) = 0.497\%$

If we assume for this example that this represented the increase in oxygen for an 8 hour period, then a 24 hour period would be represented by a 1.49% increase. The final permeation rate was arrived at by performing the following calculation:

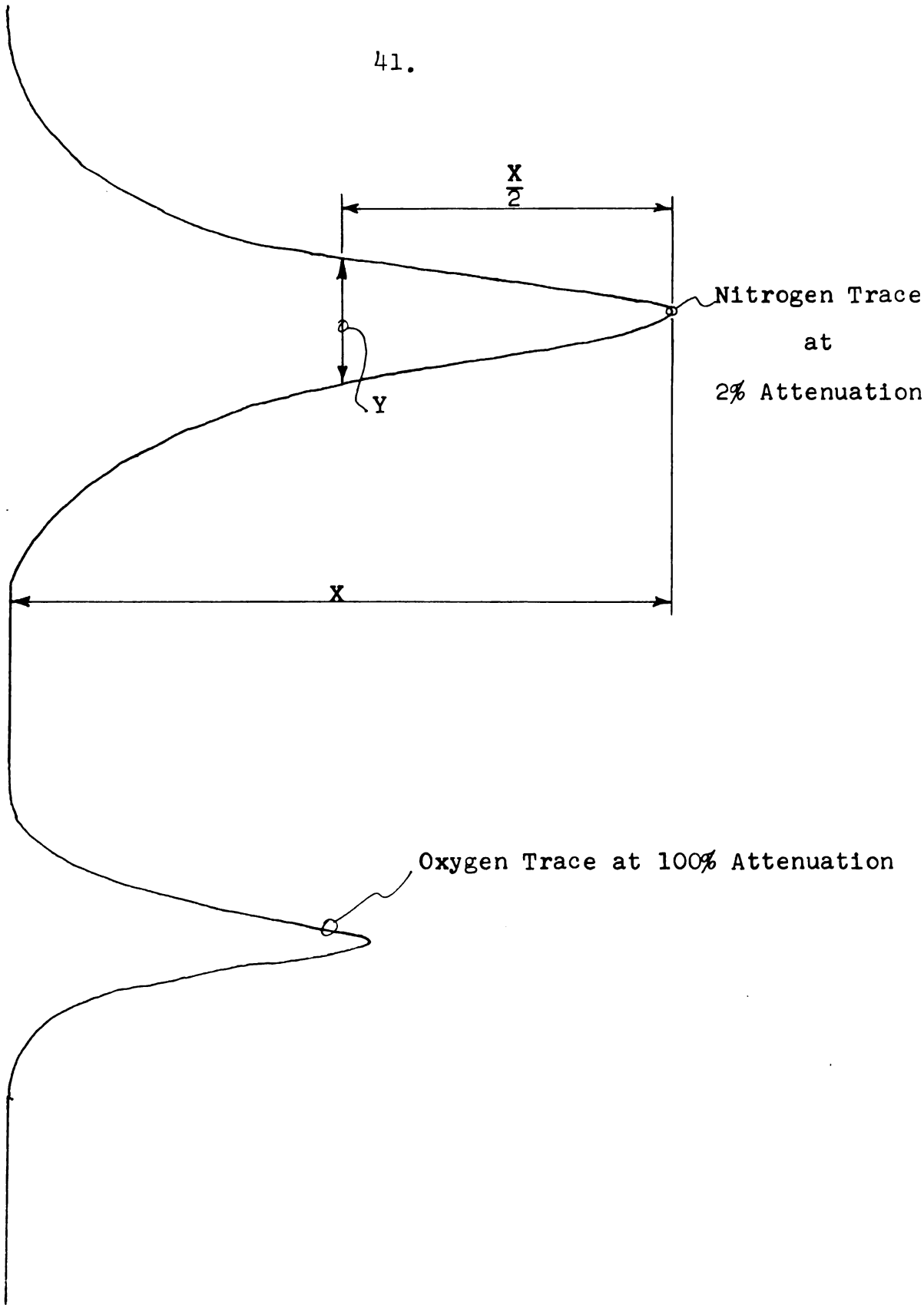

Figure 10

Recorder Trace of Partitioner Output

$$\text{Permeation rate}(\text{ml}/24\ \text{hr.}\text{-}\text{m}^2) = \frac{(\%\text{increase in } O_2/24\ \text{hr.})(20.59)}{0.015606}$$

$$\frac{(0.0149)(20.59)}{0.015606} = 19 \text{ for this example}$$

Appendix IV

Leak Test Method of Heat Seal Integrity

As described in Experimental Equipment and Procedures, it was necessary to leak test samples of heat seals prior to permeability testing. The leak test method is shown in Figure 11.

A sample of material with overlap or fin seal was placed in the Davis Cell. Valve B was closed and nitrogen was allowed to flow into the bottom half of the cell. The pressure differential created by the flowing nitrogen filling the bottom volume of the cell forced the glycerine in the manometer to react in the manner shown in Figure 11.

When the two levels of glycerine differed by approximately $1\frac{1}{2}$ to 2 inches, the metering valve was closed. This sealed the bottom half of the cell between the metering valve and valve B. If no leaks were present, the respective glycerine levels remained the same. If pinholes were present in the heat seal, the glycerine would immediately tend to level out. This was due to loss of pressure caused by the flowing of nitrogen out of the bottom half through the pinholes in the heat seal into the open top half of the cell. Any pinhole was immediately detected by utilizing this method; all pinholes were recognized by the rapid leveling of the respective glycerine levels within a maximum of three minutes.

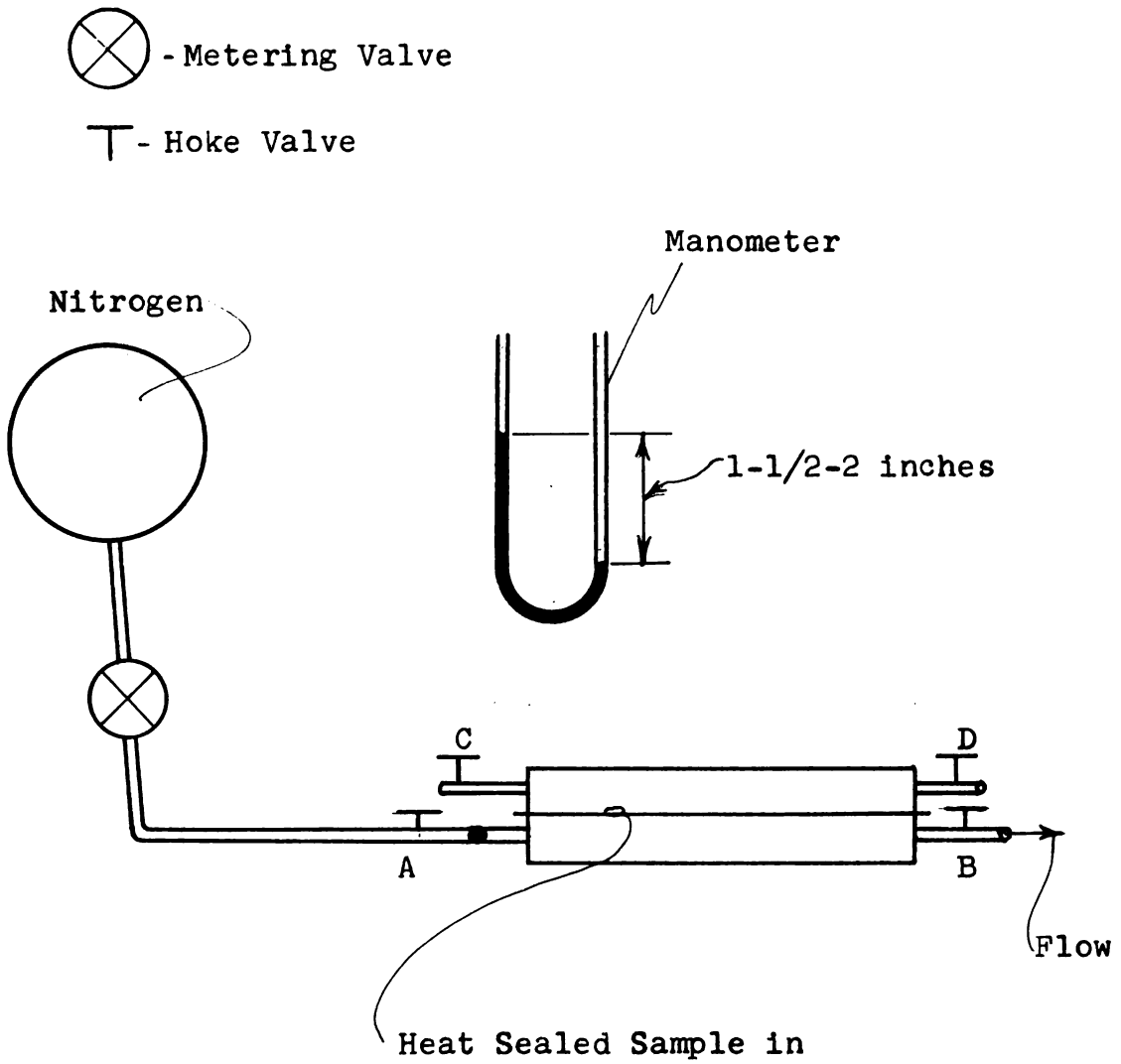

Figure 11

Leak Test Method of Heat Seal Integrity

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